

Document Number
TRACOR 65-224-U

*Top copy
being
checked*

466383

QUARTERLY STATUS REPORT NO. 9

1 January - 31 March 1965

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS
OF N-F COMPOUNDS

Contract No. Nonr-4054(00)

Research Project No. RR001-06-02

ARPA Order No. 399, Program Code No. 2910

June 30, 1965

DD
JUL 21
JISIA

TRACOR

CATALOGED BY: DDC
AS AD NO.

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

TRACOR, INC.

1701 Guadalupe St. Austin, Texas 78701

QUARTERLY STATUS REPORT NO. 9

1 January - 31 March 1965

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS OF N-F COMPOUNDS

Contract No. Nonr-4054(00)

Research Project No. RR001-06-02

ARPA Order No. 399, Program Code No. 2910

June 30, 1965

Prepared by:

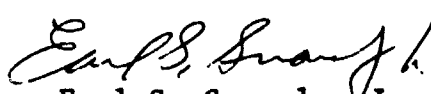
Larry G. Spears

Larry D. Fiel

Wallace E. Harrell, Jr.

Margaret H. Northrup

Approved by:



Earl S. Snavely, Jr.
Director of Chemical
Research

Reproduction in whole or in part is permitted for any purpose of
the United States Government.

TABLE OF CONTENTS

	<u>Page</u>
LIST OF ILLUSTRATIONS	iii
LIST OF TABLES	iv
ABSTRACT	v
I. INTRODUCTION AND BACKGROUND	1
II. EXPERIMENTAL APPARATUS	2
III. ANALYSIS OF ELECTROLYSIS PRODUCTS	7
IV. WORKING ELECTRODES	11
V. DISCUSSION	20
VI. FUTURE WORK	21
REFERENCES	22

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	GAS AND LIQUID SAMPLING SYSTEM	3
2	CATHODE COMPARTMENT MOUNTED IN CELL LID	4
3	ELECTROLYTIC CELL ELECTRODE CONFIGURATION - TOP VIEW	5
4	WEIGHT CHANGES OF NICKEL ALLOYS IN AHF AT OPEN CIRCUIT	13
5	WEIGHT LOSSES ON NICKEL IN HF + 0.012M NaF + H ₂ O AT 6.0 v	16
6	INITIAL POLARIZATION CURVES FOR NICKEL IN HF + 0.01M NaF + H ₂ O	17
7	POLARIZATION CURVES FOR NICKEL IN HF + 0.01M NaF + H ₂ O	19

TRACOR, INC.

1701 Guadalupe St. Austin, Texas 78701

LIST OF TABLES

<u>Table</u>		<u>Page</u>
I	PERFORMANCE OF G. C. COLUMNS FOR N-F COMPOUNDS	10
II	WEIGHT LOSS RATES OF NICKEL ALLOYS AT OPEN CIRCUIT AND AT CONSTANT-CURRENT ANODIC BIAS IN AHF	12
III	WEIGHT LOSSES OF NICKEL IN HF + 0.012M NaF + H ₂ O	15

Abstract

Work during the past quarter was devoted to the development of analytical methods for quantitative determination of N-F compounds and F_2 , and to the investigation of possible new electrode materials. Weight loss measurements, at open circuit and at constant anodic potential were performed on Incoloy 800, Inconel 600, and Ni-Span-C 902. Weight loss rates generally increased with increasing iron content and decreasing nickel content on these alloys. Weight loss measurements and polarization studies were performed on nickel in HF at various water concentrations. The addition of at least $10^{-2}\%$ H_2O reduced corrosion rates of nickel at 6.0 volts. The polarization curves showed a peak dependent on the water concentration on the initial run and a water-dependent plateau in the successive curves.

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS OF N-F COMPOUNDS

I. INTRODUCTION

Anodic polarization curves of Monel in AHF solutions of NH_4F have not revealed any concentration-dependent plateaus which indicate a stepwise fluorination of the ammonium ion. This shows that the fluorination of ammonium ions proceeds by chemical reaction rather than by an electrochemical mechanism. However, the investigation is proceeding to determine if ions such as NH_3F^+ , NH_2F_2^+ , NHF_3^+ or NF_4^+ exist as intermediates in the fluorination of NH_3 in AHF. The possibility that NF_3 is oxidized at the anode to form the transient species $\cdot\text{NF}_3^+$ which may react with $\cdot\text{F}$ to form NF_4^+ is also being investigated.

At present, the electrochemical studies are being supplemented with IR spectrophotometric and gas chromatographic (GC) analysis to determine the relative contribution of current toward the formation of each species evolved at the anode (e.g., $\cdot\text{F}_2$, NF_3 , etc.). A quantitative analysis of each product formed is necessary to establish the mechanism for the fluorination of NH_3 in AHF.

Several new electrode materials were investigated during the past quarter in an attempt to find a better electrode material. Those investigated were: nickel, Inconel 600, Incoloy 800, and Ni-Span-C 902. Monel and nickel electrodes are currently being used in electrochemical fluorination studies as they have the best properties of the metals evaluated in past experiments.

II. EXPERIMENTAL APPARATUS

A gas and liquid sampling system has been installed to allow accurate sampling of the anodic and cathodic gases and the NH_4F -AHF solution during electrolysis (see Figure 1). A porous Teflon (TFE) cathode compartment has been installed to separate the anodic and cathodic gas products (see Figure 2). The electrode configuration in the electrolytic cell is shown in Figure 3.

The procedure for gas sampling is as follows:

- (1) All the 1/8" Monel tubing leading from the 1/8" Hoke needle valve, which connects to the anodic gas line, to and including the large Monel bomb, is evacuated.
- (2) The anode condenser valve is closed and the cathode condenser valve opened. (If the cathode gas was being analyzed the reverse would be done.)
- (3) The electrolysis is started. After periods of approximately five minutes each, the needle valve is opened slightly to allow the cell pressure to decrease back to atmospheric pressure and thus collect the anodic gas products in the large bomb. The Monel pressure gauge indicates the amount of sample collected. Once the desired amount of gas is collected the bomb is sealed off and the electrolysis stopped.
- (4) One or both of the smaller Monel bombs and/or the IR gas cell is then evacuated. Then a sample of the collected gas is introduced into

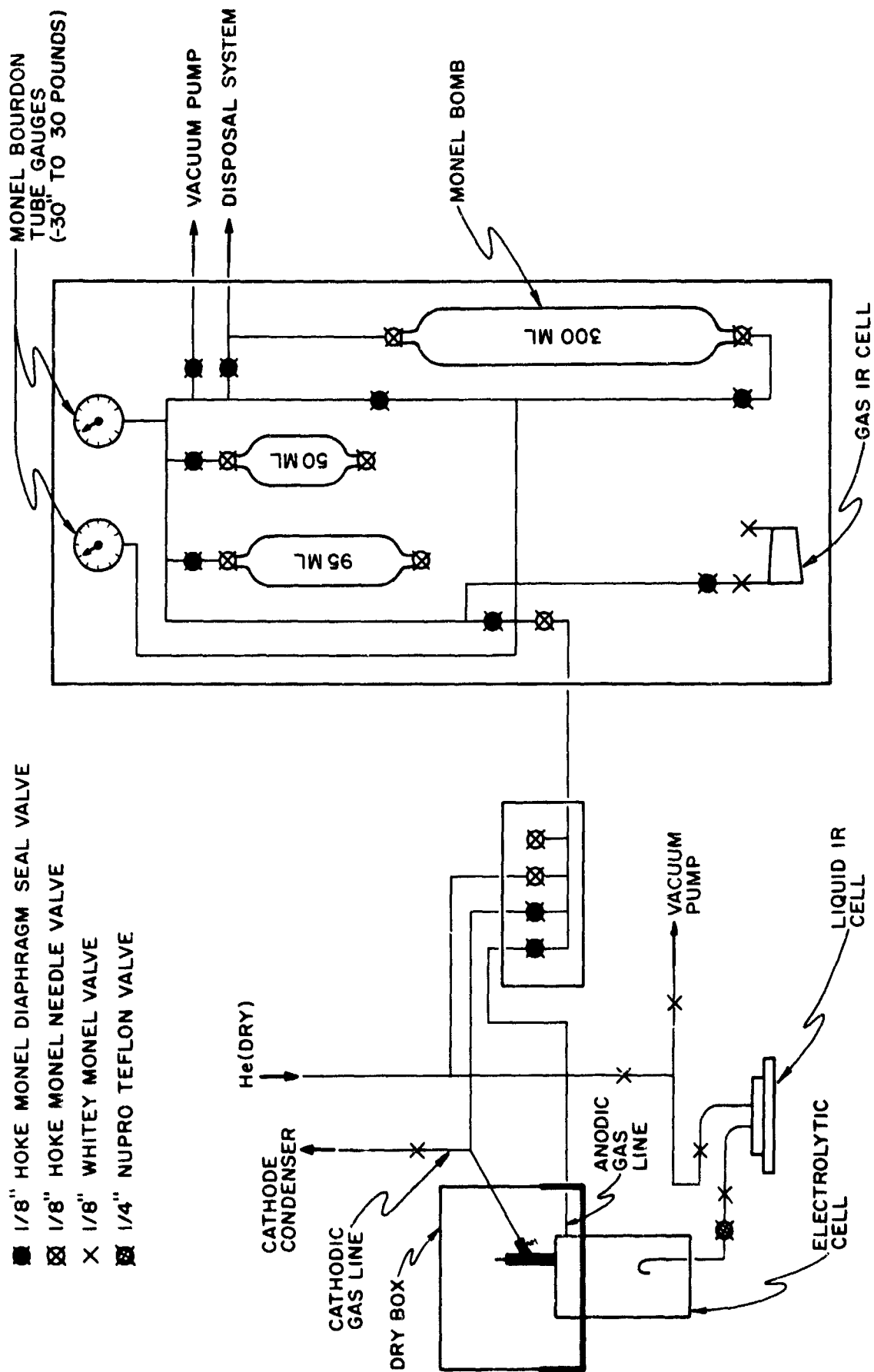


Fig I - GAS AND LIQUID SAMPLING SYSTEM

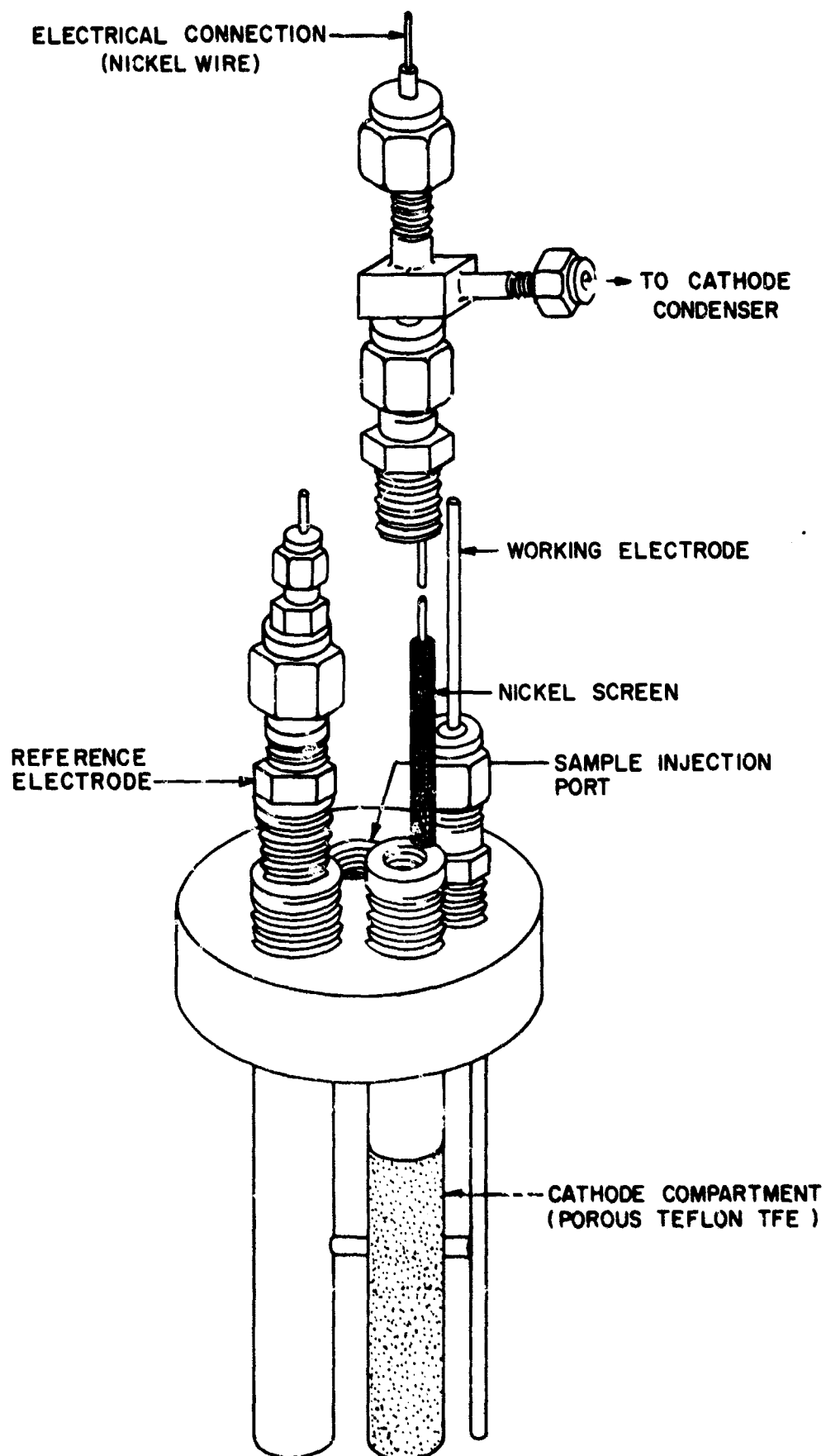


Fig. 2 - CATHODE COMPARTMENT MOUNTED IN
CELL LID

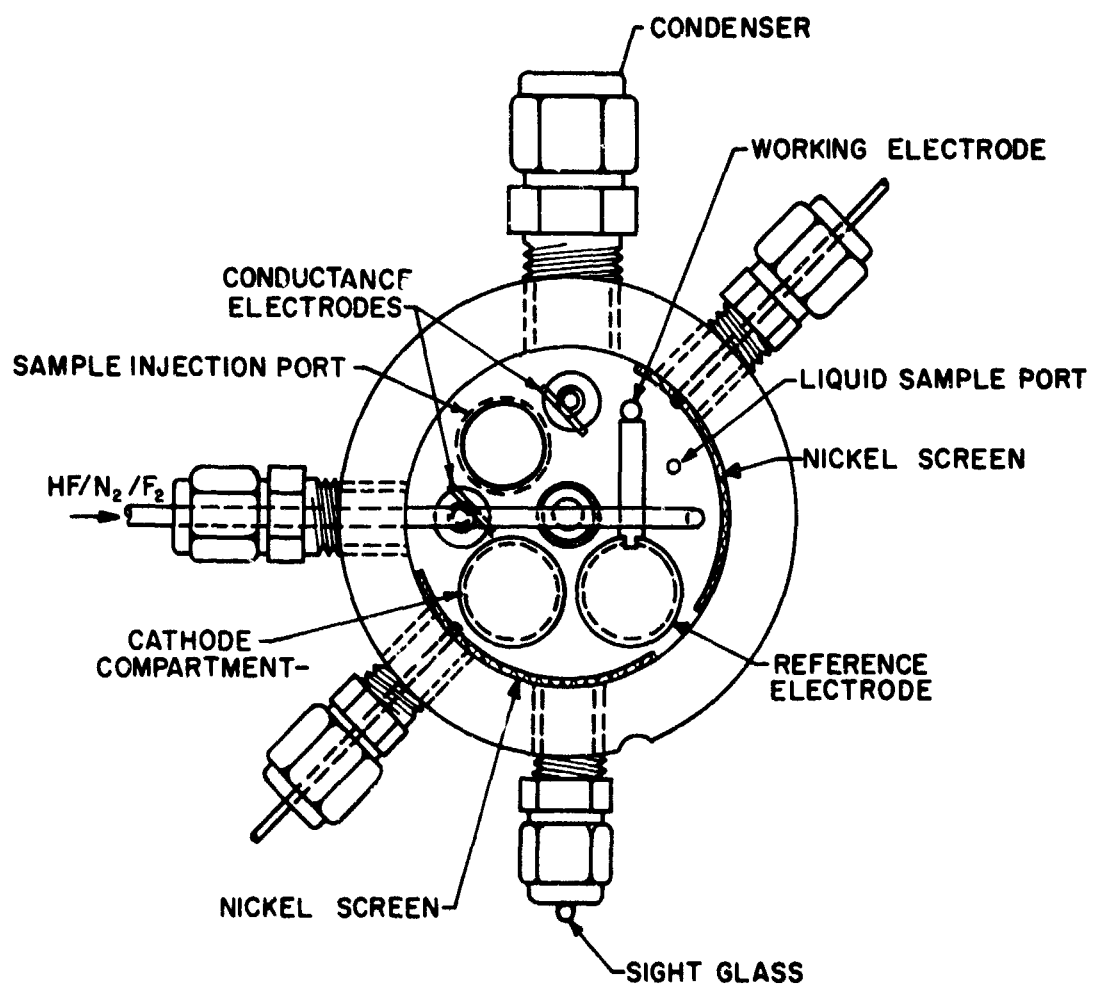


Fig. 3 - ELECTROLYTIC CELL ELECTRODE CONFIGURATION
TOP VIEW

the desired container for analysis. Samples from the smaller bombs are used for GC, NMR, and mass spectral analysis. Dry helium is used to pressurize any part of the sampling system or to flush out any part of the system.

The procedure for liquid sampling is as follows:

- (1) Dry helium is used to flush out the Kel-F liquid sample loop in the base of the cell.
- (2) A vacuum is pulled up to the 1/4" Nupro Teflon valve and then the 1/8" Whitey valve nearest the vacuum pump is closed.
- (3) Then the Teflon valve is opened to allow the liquid cell to fill. After several seconds the Teflon valve is closed and the other Whitey valve on the IR cell is closed.

A liquid IR cell was developed for use with liquid AHF. The cell is vacuum and pressure tight and should be sufficient for any low temperature studies.

III. ANALYSIS OF ELECTROLYSIS PRODUCTS

Much work during this past quarter has been devoted to developing IR and GC methods for analysis of the electrolysis products.

An attempt was made to analyze any electrolysis products remaining in solution after electrolyzing a 0.10M solution of NH_4F in AHF for four hours. This was not successful due to the reaction of the sample with the Kel-F grease which was needed to seal the commercial liquid cell used at that time. The Kel-F grease reacted to form a black residue. A new liquid IR cell has been developed in which the sample contacts only Irtran-2, Monel, and Teflon. This cell will be used at subambient temperatures in order to approximate the conditions in the electrolytic cell. A more accurate description of this cell will be given in the next quarterly report. An attempt was also made to analyze the anodic volatile products from this electrolysis. However, the Irtran-2 windows in the IR gas cell reacted to give a complicated spectrum between 800 to 2500 cm^{-1} . Due to the lack of a better gas cell window material, it will be necessary to analyze the volatile electrolysis products at subambient temperatures. However, Irtran-2 does not react in liquid HF at -19°C . A highly polished piece of Irtran-2 was placed in AHF for 48 hours and showed no weight change or dullness. Results from the GC analysis of these products were also erroneous since the Kel-F oil on the shredded Teflon packing apparently reacted with the sample to give a very poor chromatogram. Peak separation for this column is also very poor.

Investigation of gas chromatographic methods for determining F_2 and N-F compounds has been concentrated in two areas. The first is a conventional approach employing either a coated support, an uncoated active solid, or a liquid modified solid that would be

selective enough to separate components of interest and still remain unreactive to them. The second approach is to use sub-ambient distillation and thermal conductivity to detect the components. Due to the reactivity of F_2 towards Monel and stainless steel, it is necessary to passivate all the metal parts of the gas chromatograph that come in contact with the samples. Also, the normally used stainless steel detector block with tungsten filaments was replaced by a nickel detector block with Teflon-coated tungsten filaments. This was necessary since the uncoated filaments deteriorated rapidly when used with F_2 thus yielding irreproducible results. The Teflon coated filaments have shown no change after repeated exposure to fluorine and fluorine-containing compounds.

The selection of a good column packing proved to be a difficult task. Silica gel will separate air and NF_3 best, but experiments showed that F_2 reacts with silica gel, thus eliminating any quantitative measurements for F_2 if it is used as a column packing. Molecular sieves were also rejected as a column packing due to the reactivity between F_2 and metal oxides such as aluminum and magnesium oxide. Shredded Teflon (30-50 mesh) and Kel-F molding powder (30-50 mesh) were tried as supports while #10 and #40 Kel-F oils and #13-21 Halocarbon oil were investigated as liquid phases. Even when immersed in dry ice-acetone baths, these columns gave poor retention times and were not reproducible. These columns have also been rejected.

Reaction chromatography was also considered. An example of this is the quantitative analysis for F_2 . A fluorine-containing sample is injected into a precolumn filled with NaCl to form $NaF + Cl_2$. Then the chlorine is directed into a GC column for analysis. This was rejected because compounds such as N_2F_2 (a possible anodic product) would also react with NaCl.

Based on preliminary experiments, the best approach is subambient distillation or cryogenic programming to separate the volatile products and identifying them by gas chromatography and infrared spectroscopy. This method will also allow the determination of the boiling point for any unknown compounds. An F & M Chromatograph, Model 720, with a nickel block and Teflon coated filaments and a 0-1 mv strip-chart recorder with a one-second full-scale response is used for this method. A coiled, 16', 1/4" o.d. Monel tube (previously passivated with F_2) placed inside a large Dewar is used as a column and liquid nitrogen as a coolant. Helium, 99.95% pure, is used as the carrier gas. Before it enters the GC column it passes through a 10' x 1/4" o.d. copper tube filled with molecular sieves and then through a 10' x 1/4" o.d. copper tube immersed in a liquid nitrogen bath. Before a sample from one of the sample bombs is allowed to enter the sample loop, the loop is evacuated to at least 1 cm of Hg, and then the loop is pressured up to atmospheric pressure with the sample. Six minutes after the sample is injected into the column the liquid N_2 is drained through a valve in the bottom of the Dewar, and a heating rod located in the center of the coil (1000 watts at 35 volts) is turned on. Nitrogen gas is then allowed to flow through the valve in the bottom of the Dewar at a rate of 150 cc/min for uniform heating. Two Chromel-Alumel thermocouples, one in an ice bath and the other next to the GC column, and a student potentiometer are used to determine the change in temperature of the column. With the parameters given above, a linear heating rate of $4^\circ C/min$ from -196° to $20^\circ C$ is obtained. Data from this method and the chromatographic columns are given in Table I.

TABLE I
PERFORMANCE OF G. C. COLUMNS FOR N-F COMPOUNDS

COLUMN AND CONDITIONS	(THEORETICAL PLATES PER COLUMN) ON NF ₃	COLUMN EFFICIENCY HE/P (cm)	RETENTION TIMES, MIN.				COMMENTS	
			AIR	F ₂	NF ₃	N ₂ F ₄		
Column 1 16-ft. - 1/4" O.D., Monel tube, liq. N ₂ starting temperature, 50 cc/min. He flow. Program rate = ~ 3.0°C/min.	903	0.54	7.5	7.7	24.2	31	72.4	This column and parameters still under development. Peaks are leading and carrier gas needs purifying.
Column 2 20-ft. - 1/4" O.D., Monel column packed with 50% Halocarbon Oil 13-21 on 30-50 mesh Kel-F molding powder. 65 cc/min. He flow. 40°C.	100	6.1	0.8	0.8	1.0	-	2.4	Not enough resolution between F ₂ and NF ₃ .
Column 3 10-ft. - 1/4" O.D., aluminum column packed with 30-60 mesh Silica gel, 60°C, 60 cc/min. He flow.	345	0.89	3.7	-	6.5	-	-	Peaks are well defined and sharp.
Column 4 50-ft. - 1/8" O.D., Kel-F tubing, packed with 50% Halocarbon Oil 13-21 on 30-50 mesh Kel-F molding powder, 45 cc/min. He flow. -72°C.	1410	1.1	7.0	7.0	7.5	-	-	Not enough resolution between F ₂ and NF ₃ .
Same as Column 4, ambient temp.	503	3.3	5.1	-	5.6	-	-	Sharp, Gaussian peaks.

IV. WORKING ELECTRODES

Work on inert anode materials was concentrated on nickel alloys since these possess the desirable characteristics (low corrosion rates in AHF and formation of a passive conducting film) of a good working electrode. In view of this, Inconel 600(1), Incoloy 800(1), Ni-Span-C 902(1), and nickel(2) were investigated during the past quarter for possible use as an anode material in AHF.

A. Nickel Alloys

Corrosion measurements were made on Inconel 600, Incoloy 800, and Ni-Span-C 902 in AHF at open circuit and at anodic bias. Weight loss rates are shown in Table II and the periodic weight losses are shown in Figure 4.

In comparing the three alloys, it is noted that corrosion rates generally increase with increasing iron content and with decreasing nickel content.(1) These alloys did not show sufficient promise for use as anodes in AHF to warrant further study, and future investigations of anode materials will be concentrated on nickel.

B. Nickel

Corrosion studies were conducted on nickel electrodes in HF with NaF added as a supporting electrolyte at various water concentrations. Increased resistance to attack in the presence of water was shown previously by Donohue and Nevitt (3). Open circuit weight loss measurements were made on nickel electrodes in $\text{HF} + 0.01\text{M NaF} + \text{H}_2\text{O}$. Water concentrations of $\leq 10^{-3}$ wt.% to 10^{-1} wt.% were used. Weight losses were low ($\leq 0.2 \text{ mg/cm}^2$) for all water concentrations for periods of time up to nine hours. The weight

TABLE II

WEIGHT LOSS RATES OF NICKEL ALLOYS AT OPEN CIRCUIT
AND AT CONSTANT-CURRENT ANODIC BIAS IN AHF

<u>Metal</u>	<u>Current Density ($\mu\text{a}/\text{cm}^2$)</u>	<u>Time (hrs)</u>	<u>Weight Loss Rate ($\text{mg}/\text{cm}^2/\text{hr}$)</u>
Inconel 600	open circuit	16	0.002
Inconel 600	40^{a}	24	0.013
Inconel 600	600^{b}	2	0.535
Incoloy 800	open circuit	16	0.005
Incoloy 800	40^{a}	24	0.028
Incoloy 800	600^{b}	2	0.610
Ni-Span-C 902	open circuit	16	0.006
Ni-Span-C 902	40^{a}	24	0.046
Ni-Span-C 902	600^{b}	2	0.780

a -- Below Fluorine evolution potential

b -- Above Fluorine evolution potential

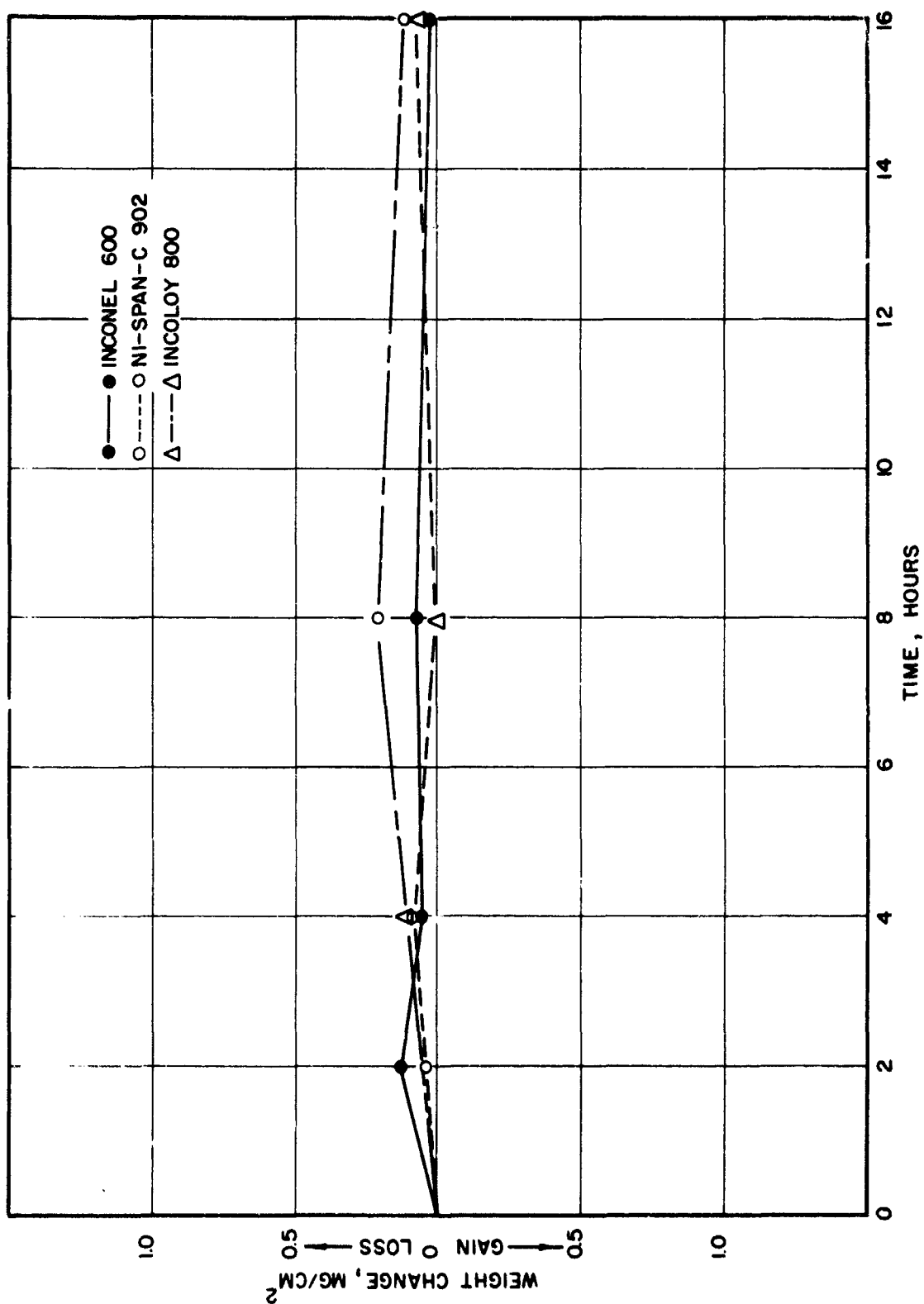


Fig. 4- WEIGHT CHANGES OF NICKEL ALLOYS IN AHF AT OPEN CIRCUIT.

losses approach a constant value with time, indicating formation of a passive film, which protects the electrode from further corrosion.

Anodic weight losses at 1.0 v, 3.0 v and 6.0 v are tabulated in Table III. It is noted that the addition of a small amount of water, on the order of 10^{-2} wt%, causes a large decrease in the corrosion rate at 6.0 v (see Figure 5). This effect has been observed on Hastelloy-F in our laboratory (4) and on nickel by Donohue and Nevitt (3). Weight losses at 1.0 v and 3.0 v are small and show no significant changes with water concentration.

A change in the character of the film was noted with increasing water concentration, particularly at 6.0 v. In HF with $\leq 10^{-3}$ % H_2O , the film was thick and crusty, and weakly adhering. This film was light green in color and was easily washed off in hot water. Large weight losses were observed with this type of film. At higher water concentrations, a dark (gray-black), thin, tightly adhering film was formed on the electrode. This film was difficult to remove without attack on the nickel itself, which probably accounts for the discrepancies in the measured and calculated weight losses. A cleaning process using cathodic protection of the nickel is being developed, which should facilitate more accurate weight measurements.

Anodic polarization curves were run on nickel electrodes in HF + 0.012M NaF with water concentrations from $\leq 10^{-3}$ % to 10%. The initial polarization curves, begun after the electrode was in solution long enough for the open circuit potential to reach equilibrium, are shown in Figure 6. (It is noted that the open circuit potential is shifted cathodically as the water concentration is increased as indicated by the arrows.) A sharp spike appeared at 0.0 v after the first water addition. The intensity of the peak increased as the water concentration was increased.

TABLE III

WEIGHT LOSSES OF NICKEL IN HF + 0.012M NaF + H₂O

H ₂ O Concentration (wt%)	Potential (volts) vs Hg/Hg ₂ F ₂	Time (hrs)	Weight Loss (mg/cm ²)		
			Calc. from defilmed wt.	Calc. from Gain*	Calc. from Charge*
0.001	1.0	17	-	1.050	0.055
0.02	1.0	24	0.102	0.132	0.036
0.05	1.0	24	0.810	1.2	0.108
0.1	1.0	21	0.04	0.21	0.081
1.0	1.0	23	0.108	-	0.180
10.0	1.0	24	0.080	0.015	0.039
0.001	3.0	16	-	0.132	0.480
0.02	3.0	16	0.048	0.090	1.10
0.05	3.0	16	0.050	0.700	0.720
0.1	3.0	16	-	0.40	0.708
1.0	3.0	16	0.270	-	0.864
10.0	3.0	16	0.090	-	0.680
0.001	6.0	0.06	1.73	3.01	2.34
0.02	6.0	0.06	0.14	0.42	2.64
0.05	6.0	0.06	0.13	0.69	3.0
0.1	6.0	0.06	0.21	0.43	2.9
1.0	6.0	0.10	0.16	0.22	4.5
10.0	6.0	0.16	0.12	0.027	1.8

* Assuming formation of NiF₂

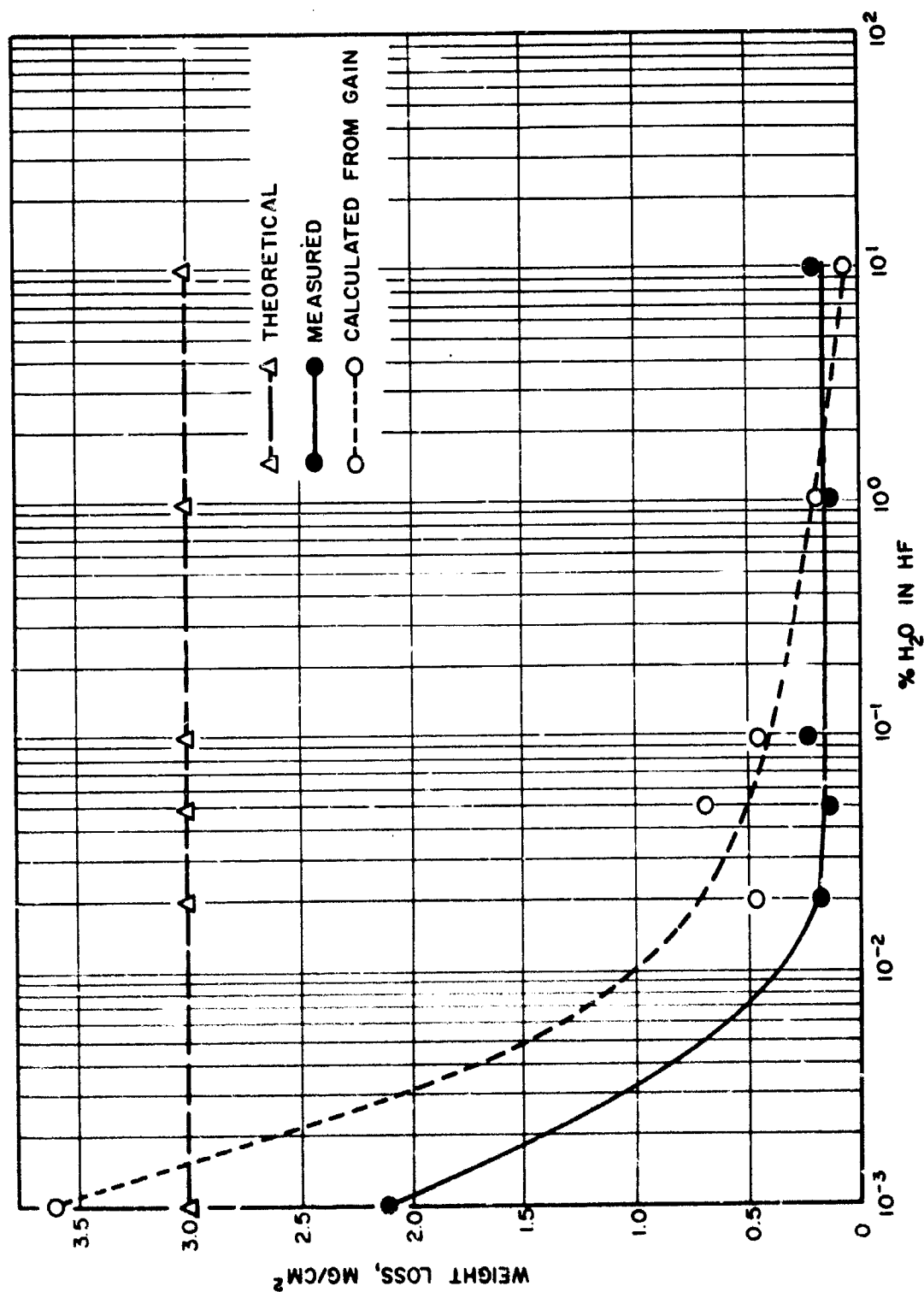


FIG. 5—WEIGHT LOSSES ON NICKEL IN HF + 0.012M NaF + H₂O AT 6.0 V

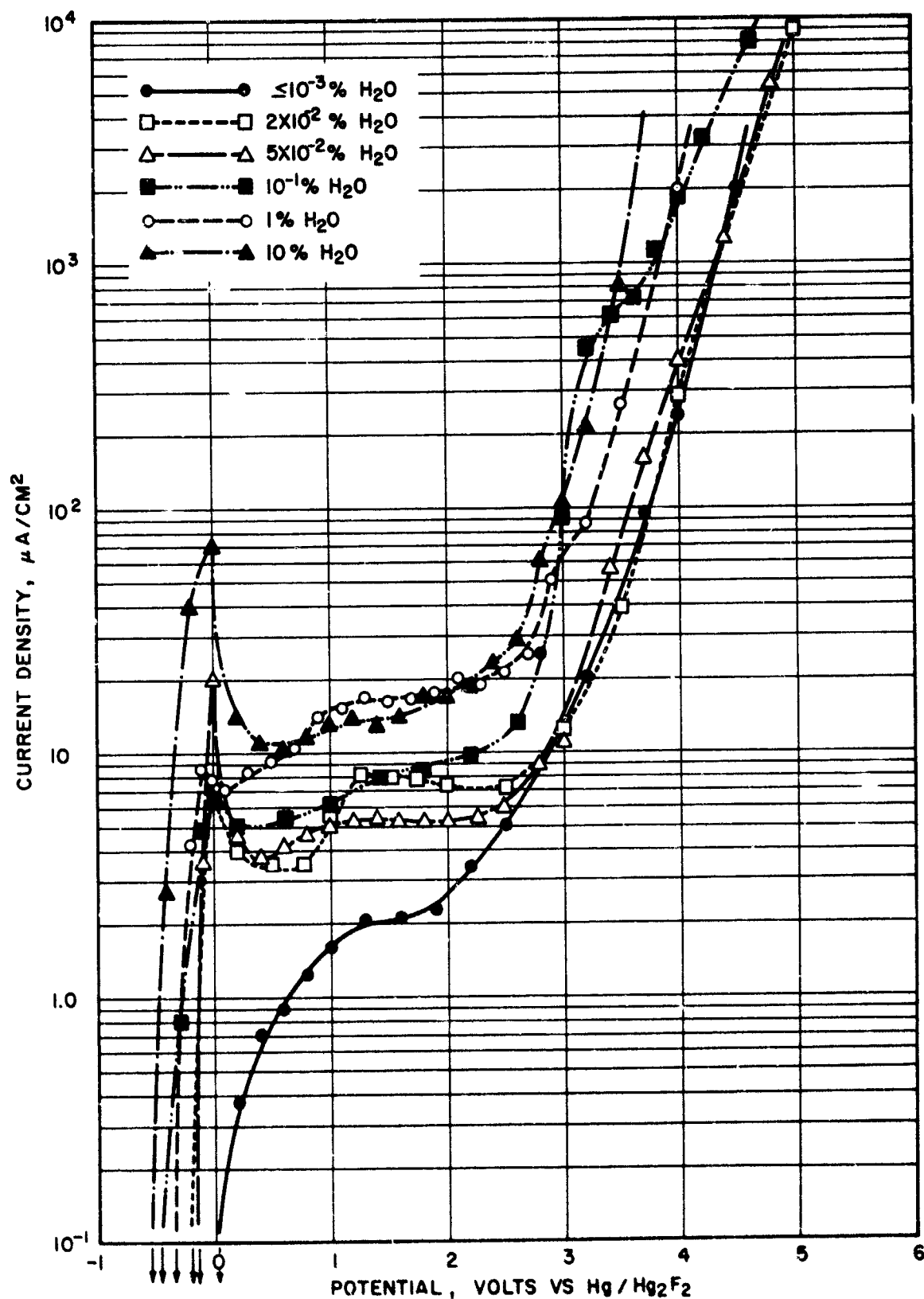


Fig.6-INITIAL POLARIZATION CURVES FOR NICKEL
IN HF + 0.01M NaF + H₂O

This peak has been observed on many metals in the course of our investigations and is probably related to the formation of a metal-metal fluoride film. Successive polarization curves on the same electrode (Figure 7) do not exhibit this peak, indicating that a passive film is formed on the initial polarization run. However, these curves do show an increase in the current density with water concentration in the potential range of 0.0 v to 3.0 v (see Figure 8). It has been ascertained by weight loss measurements that this increase is not due to metal dissolution.

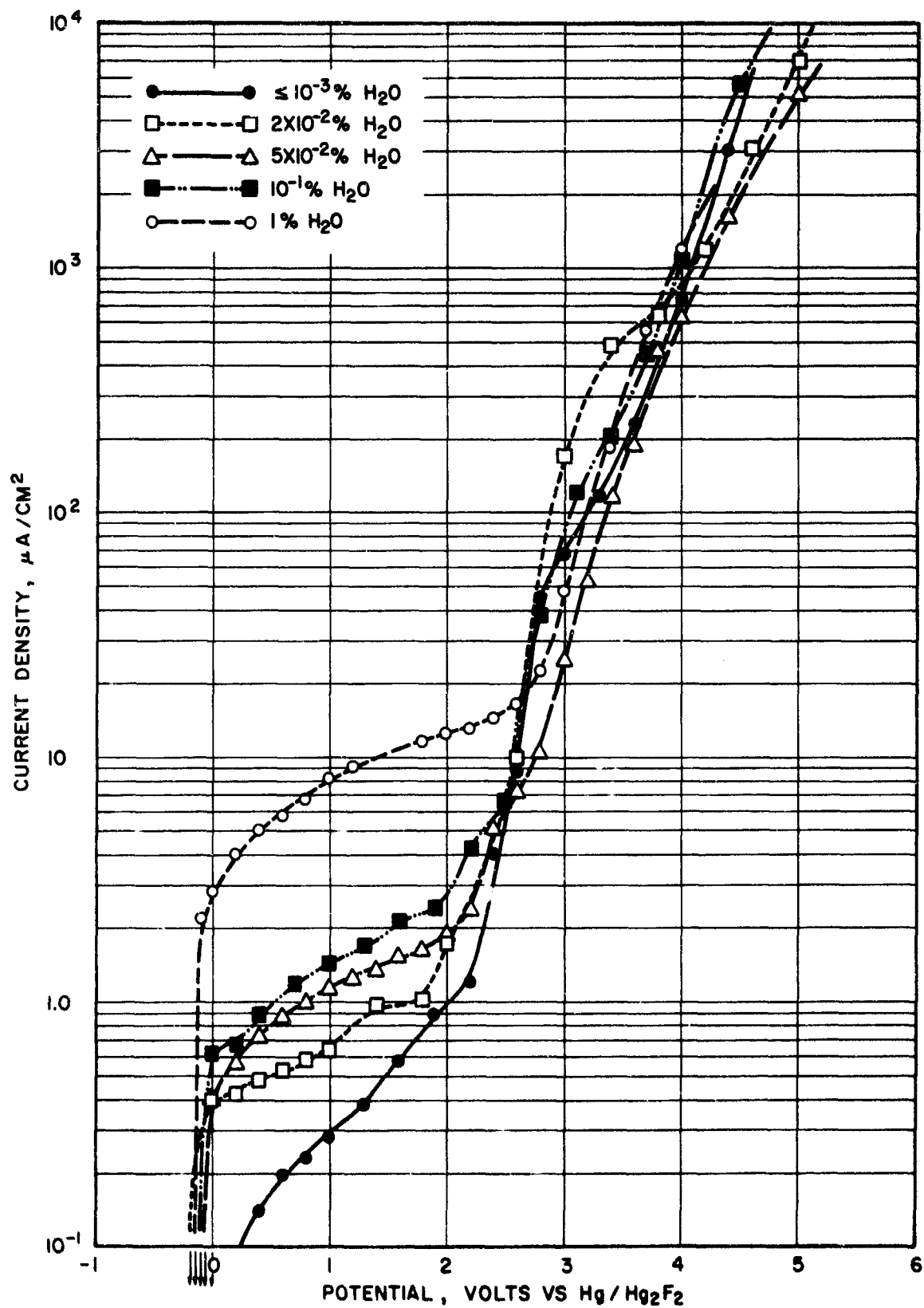


Fig. 7 - POLARIZATION CURVES FOR NICKEL
IN HF + 0.01M NaF + H₂O

V. DISCUSSION

Results to date indicate that it is necessary to perform the IR gas analysis at subambient temperatures and the IR liquid analysis at temperature below the boiling point of HF (19°C). Due to the lack of a suitable material to pack the GC columns, the volatile electrolysis products will be analyzed on the gas chromatograph using a 16-foot long, 1/4" diameter coiled Morel tube immersed in liquid nitrogen initially and cyrogenically programmed to the desired temperature.

The behavior of a nickel anode in HF is very much dependent on the water concentration of the solution. A passive behavior is observed when the water concentration reaches approximately $10^{-2}\%$.

VI. FUTURE WORK

The studies on the electrochemical fluorination of NH_4F at various anodic potentials and identifying the products formed by IR and GC analysis will be continued. Cryogenic programming will be used for the GC analysis. Cathodic stripping curves, galvanostatic polarization curves, and further weight loss studies will be performed on nickel in HF to determine the role of anode material during electrochemical fluorination.

REFERENCES

1. Huntington Alloy Products Division, The International Nickel Company, Inc., Huntington, West Virginia.
2. Nickel Analysis:

Cobalt	-	0.060%
Copper	-	0.006%
Iron	-	0.010%
Sulfur	-	0.015%
Carbon	-	0.008%
Nickel	-	99.901%
3. J. A. Donohue, and T. D. Nevitt, ARPA Propellant Contractor's Conference, Chicago, Illinois, April 13-15, 1964.
4. E. S. Snavely, Jr., et al, "Electrochemical Studies in the Synthesis of N-F Compounds," Quarterly Status Report No. 7, TRACOR, Inc. Document No. 65-103-U, dated January 8, 1965, p. 12.

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)

TRACOR, Inc.
1701 Guadalupe
Austin, Texas 78701

2a. REPORT SECURITY CLASSIFICATION

UNCLASSIFIED

2b. GROUP

3. REPORT TITLE

QUARTERLY STATUS REPORT NO. 9
1 JANUARY - 31 MARCH 1965
ELECTROCHEMICAL STUDIES IN THE SYNTHESIS OF N-F COMPOUNDS

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Quarterly

5. AUTHOR(S) (Last name, first name, initial)

Spears, Larry G.
Fiel, Larry D.
Harrell, Wallace E., Jr.

Northrup, Margaret H.
Snively, Earl S., Jr.

6. REPORT DATE

30 June 1965

7a. TOTAL NO. OF PAGES

27

7b. NO. OF REFS

4

8a. CONTRACT OR GRANT NO.

Nonr-4054(00)

b. PROJECT NO.

RR001-06-02

c. ARPA Order No. 399

d. Program Code No. 2910

9a. ORIGINATOR'S REPORT NUMBER(S)

65-224-U

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. AVAILABILITY/LIMITATION NOTICES

"Qualified requesters may obtain copies of this report from DDC."

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

Department of the Navy
Office of Naval Research
Power Branch, Washington, D.C. 20360

13. ABSTRACT

Work during the past quarter was devoted to the development of analytical methods for quantitative determination of N-F compounds and F_2 , and to the investigation of possible new electrode materials.

Weight loss measurements, at open circuit and at constant anodic potential were performed on Incoloy 800, Inconel 600, and Ni-Span-C 902. Weight loss rates generally increased with increasing iron content and decreasing nickel content on these alloys. Weight loss measurements and polarization studies were performed on nickel in HF at various water concentrations. The addition of at least $10^{-2}\%$ H_2O reduced corrosion rates of nickel at 6.0 volts. The polarization curves showed a peak dependent on the water concentration on the initial run and a water-dependent plateau in the successive curves.

DD FORM 1473
1 JAN 64

UNCLASSIFIED

Security Classification

UNCLASSIFIED
Security Classification

14.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
Quantitative Chemical Analysis Electrochemistry Electrode Materials IR Spectrophotometric Analysis Gas Chromatographic Analysis N-F Compounds Fluorine Compounds							

INSTRUCTIONS

1. **ORIGINATING ACTIVITY:** Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization (*corporate author*) issuing the report.
- 2a. **REPORT SECURITY CLASSIFICATION:** Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.
- 2b. **GROUP:** Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.
3. **REPORT TITLE:** Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.
4. **DESCRIPTIVE NOTES:** If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.
5. **AUTHOR(S):** Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.
6. **REPORT DATE:** Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.
- 7a. **TOTAL NUMBER OF PAGES:** The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.
- 7b. **NUMBER OF REFERENCES:** Enter the total number of references cited in the report.
- 8a. **CONTRACT OR GRANT NUMBER:** If appropriate, enter the applicable number of the contract or grant under which the report was written.
- 8b, 8c, & 8d. **PROJECT NUMBER:** Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.
- 9a. **ORIGINATOR'S REPORT NUMBER(S):** Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.
- 9b. **OTHER REPORT NUMBER(S):** If the report has been assigned any other report numbers (*either by the originator or by the sponsor*), also enter this number(s).
10. **AVAILABILITY/LIMITATION NOTICES:** Enter any limitations on further dissemination of the report, other than those

imposed by security classification, using standard statements such as:

- (1) "Qualified requesters may obtain copies of this report from DDC."
- (2) "Foreign announcement and dissemination of this report by DDC is not authorized."
- (3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through _____."
- (4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through _____."
- (5) "All distribution of this report is controlled. Qualified DDC users shall request through _____."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. **SUPPLEMENTARY NOTES:** Use for additional explanatory notes.

12. **SPONSORING MILITARY ACTIVITY:** Enter the name of the departmental project office or laboratory sponsoring (*paying for*) the research and development. Include address.

13. **ABSTRACT:** Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

UNCLASSIFIED

Security Classification